(19)日本国特許庁（ＪＰ）　(12) 公開特許公報（Ａ）　(11)特許出願公開番号
特開2003－63894
（P2003－63894A）
(43)公開日　平成15年３月５日(2003.3.5)

(51)Int.Cl.⁷	識別記号	ＦＩ	テーマコード゛(参考)
Ｃ３０Ｂ　29/30		Ｃ３０Ｂ　29/30	Ｂ　４Ｇ０７７

審査請求　未請求　請求項の数２　ＯＬ　（全 ４ 頁）

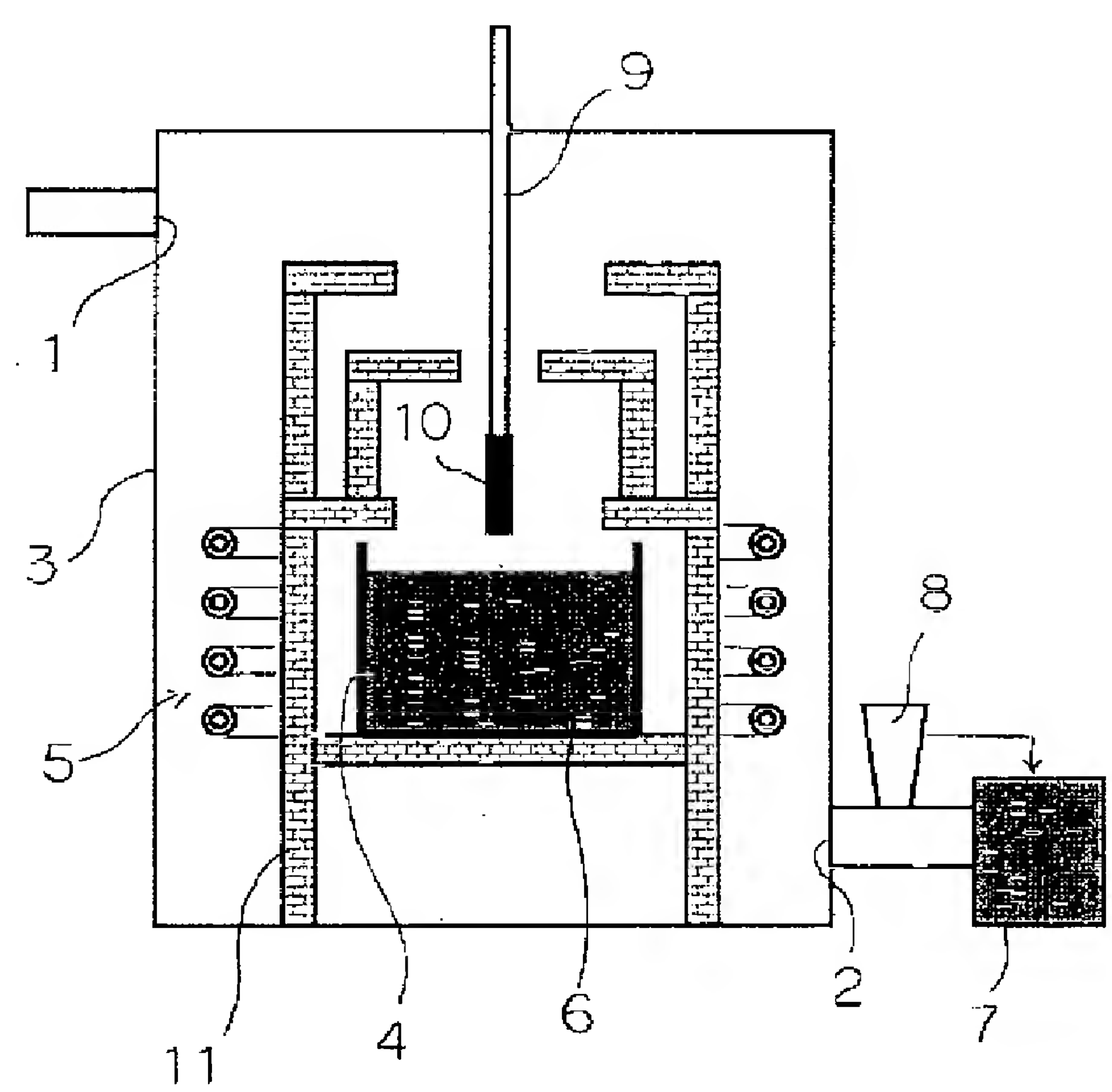
(21)出願番号　特願2001－259313(P2001－259313)
(22)出願日　平成13年８月29日(2001.8.29)

(71)出願人　000006633
京セラ株式会社
京都府京都市伏見区竹田鳥羽殿町６番地
(72)発明者　堀　健次
京都府相楽郡精華町光台３丁目５番地３号
京セラ株式会社中央研究所内

Ｆターム(参考)　4G077 AA02 BC37 CF10 EA02 EA05
EA06 EA07 EC01 ED02 HA01
PF55 PJ02

(54)【発明の名称】　タンタル酸リチウム単結晶及びその製造方法

(57)【要約】
【課題】　従来にない化学量論組成に近いコングルエント組成を有し結晶性の良好なタンタル酸リチウム単結晶、及びその製造方法を提供すること。
【解決手段】　コングルエント組成のモル比Ｌｉ／Ｔａが０．９５より大で、かつキュリー温度が６１０℃より高いタンタル酸リチウム単結晶は、酸素ガス分圧が５～２０ｈＰａ、及び不活性ガス分圧が９００ｈＰａ以下の単結晶育成炉内で、ガス圧力変動幅を１０ｈＰａ以下に制御しながらチョクラルスキー法により単結晶を育成することにより、欠陥が少ない高品質で均一組成の単結晶を育成できる。



【特許請求の範囲】

【請求項１】　コングルエント組成のモル比Ｌｉ／Ｔａが、０．９５より大で、かつキュリー温度が６１０℃より高いタンタル酸リチウム単結晶。

【請求項２】　請求項１に記載のタンタル酸リチウム単結晶をチョクラルスキー法により育成する製造方法であって、酸素ガス分圧が５～２０ｈＰａ、及び不活性ガス分圧が９００ｈＰａ以下の単結晶育成炉内で、ガス圧力変動幅を１０ｈＰａ以下に制御しながら育成単結晶を融液から引き上げるようにしたことを特徴とするタンタル酸リチウム単結晶の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、弾性表面波（ＳＡＷ）素子用基板や光学素子等として好適に用られるタンタル酸リチウム単結晶及びその製造方法に関するものである。

【０００２】

【従来の技術】従来、タンタル酸リチウム単結晶の製造には、一般にチョクラルスキー法が用いられている。この方法では、坩堝内に充填された原料を加熱溶融した後、種結晶をその融液に接触させ、しかる後に種結晶を引き上げながら温度操作により目的とする結晶径まで成長させ、その後は結晶径を一定に保つような温度操作により、種結晶と同じ方位のタンタル酸リチウム単結晶を得ることができる。

【０００３】タンタル酸リチウムの融点は約１６５０℃であるので、通常、高融点金属の高価なイリジウムが坩堝材として用いられ、高周波加熱による坩堝の加熱で原料を溶融させるが、イリジウムの消耗を抑制するために酸素濃度をできるだけ少なく（例えば、２体積％）にする必要がある。雰囲気制御は酸素と不活性ガスの混合ガスを結晶育成室である単結晶育成炉内に投入することによって行うが、単結晶育成炉排気口は開放しているため、天候により外気圧が３０～４０ｈＰａも変動すると単結晶育成炉内の圧力も同様に変動してしまう。

【０００４】

【発明が解決しようとする課題】一般に大気圧すなわち１０１３ｈＰａの条件下では、タンタル酸リチウム単結晶のコングルエント組成は、Ｌｉ：Ｔａ＝４８．３：５１．７程度であるとされているが、圧力が４０ｈＰａ程度下がるとコングルエント組成はＬｉ：Ｔａ＝４８．５：５１．５程度まで変動してしまう。

【０００５】このように、単結晶育成炉内の圧力が変動すると、コングルエント組成が変動するので、原料と育成結晶の組成ずれが発生するだけでなく、坩堝内に残留している原料組成もずれることになるので、原料を連続して充填すると大きな組成ずれが生じる。これを防止するために、その都度、原料組成の調整や原料の入れ替えが必要となる。また、大気圧下でのコングルエント組成は、Ｌｉの含有量が少ないため、結晶中のＬｉ欠陥が多く結晶性が低下し、電子部品等の基板や光学素子用として好ましくない。

【０００６】そこで本発明では、従来にない化学量論組成に近いコングルエント組成を有し結晶性の良好なタンタル酸リチウム単結晶、及びその製造方法を提供することを目的とする。

【０００７】

【課題を解決するための手段】上記課題を解決するために、本発明のタンタル酸リチウム単結晶は、コングルエント組成のモル比Ｌｉ／Ｔａが、０．９５より大で、かつキュリー温度が６１０℃より高いことを特徴とする。

【０００８】また、上記タンタル酸リチウム単結晶の製造方法は、チョクラルスキー法により単結晶を育成する製造方法であって、酸素ガス分圧が５～２０ｈＰａ、及び不活性ガス分圧が９００ｈＰａ以下の単結晶育成炉内で、ガス圧力変動幅を１０ｈＰａ以下に制御しながら育成単結晶を融液から引き上げるようにしたことを特徴とする。

【０００９】これにより、常に、欠陥が少ない高品質で均一組成の単結晶を連続して育成することが可能となる。

【００１０】

【発明の実施の形態】以下に、本発明の実施形態について模式的にあらわした図面に基づいて詳細に説明する。

【００１１】図１は本発明で用いる高周波加熱式の単結晶育成装置の断面図である。

【００１２】まず、ガスの吸気口１及び排気口２を備えた単結晶育成炉３内に、円筒状に配設された耐火セラミックス１１で包囲されたイリジウム製の坩堝４に、コングルエント組成のタンタル酸リチウム原料を充填した状態で、吸気口１より酸素（０．５～２体積％）と不活性ガス（窒素またはアルゴンを９８～９９．５体積％）の混合ガスを単結晶育成炉３に吸入する。

【００１３】次に、排気口２の近傍に設けた圧力計８により、単結晶育成炉３内の圧力を測定しながら排気ポンプ７を制御して、酸素ガス分圧が５～２０ｈＰａ、及び不活性ガス分圧が９００ｈＰａ以下の単結晶育成炉内で、ガス圧力変動幅を１０ｈＰａ以下に制御しながら単結晶育成炉内で、ガス圧力変動幅が１０ｈＰａ以下の一定圧になるように制御する。

【００１４】そして、坩堝４の周囲に設けたコイル５により、坩堝４に対して高周波加熱を行い、坩堝４内の原料を溶融して融液６とする。その後、支持棒９の先端に設けた所定方位の種結晶１０を融液６に接触させ、高周波出力を制御し加熱温度を制御しながら支持棒９を引き上げ回転させ結晶を成長させる。

【００１５】上記製造において、酸素ガス分圧が５～２０ｈＰａ、及び不活性ガス分圧が９００ｈＰａ以下の単結晶育成炉内で、ガス圧力変動幅を１０ｈＰａ以下に制

御しながら単結晶を育成する理由は、９００ｈＰａ以下のガス雰囲気中で単結晶を育成すると、一般に知られているコングルエント組成よりもＬｉリッチになることが判明し、また、組成変動を±０．０５％以内に制御するためには、圧力変動を１０ｈＰａ以下に制御する必要があることが判明したからである。

【００１６】以上により、欠陥が極めて少なく高品質で且つ均一組成の単結晶、すなわち、コングルエント組成のモル比Ｌｉ／Ｔａが０．９５より大で、かつキュリー温度が６１０℃より高く、キュリー温度のばらつきがほとんど無い、優れた単結晶を得ることができる。また、キュリー温度が高くばらつきが無いので、非線形光学定数の大きい優れた光学用途に適用が可能である。

【００１７】

【実施例】次に、本発明のより具体的な実施例について説明する。

【００１８】＜実施例１＞純度９９．９９％の炭酸リチウムと五酸化タンタルの原料を４８．８：５１．２の割合で混合した後に電気炉で焼成（雰囲気：大気）して原料組成のモル比Ｌｉ／Ｔａが０．９５３のタンタル酸リチウム原料を作製した。

【００１９】作製した原料を内径１８０ｍｍ、高さ１８０ｍｍのイリジウム製の坩堝４に充填し、引き上げ方位をＹ軸からＺ軸方向に３４°～４４°傾けた方位で、先端部の径が６ｍｍの種結晶１０とともに、図１に示す高周波加熱式単結晶育成装置にセットした。

【００２０】その後、吸気口１から酸素ガス１体積％、窒素ガス９９体積％の混合ガスを吸入し、圧力計８でガス圧力を測定しながら排気ポンプ７の排気制御を行って、単結晶育成炉３内のガス圧力が９００ｈＰａ±５ｈＰａになるように制御した。

【００２１】次いで、コイル５により坩堝４に対し高周波加熱を行い、タンタル酸リチウム原料を溶融した。原料が溶けた後、融液表面温度を融点の１６５０℃より３０～４０℃高い１６８０～１６９０℃まで昇温し、１時間保持した後、融液温度を融点まで下げた。

【００２２】次いで、種結晶１０を９．５ｒｐｍで回転させながらゆっくりと降下させ液面に接触させた後、種結晶先端部の径が５ｍｍになるように高周波出力により液面温度を制御した。そして、種結晶１０を２ｍｍ／ｈｒで回転させながら引き上げ、徐々に温度を下げて４時間程度で結晶径１０ｃｍまで結晶頭部を育成させた後に、直胴部を１１ｃｍまで育成させた。このようにして育成した結晶を融液から切り離した後、室温まで３０時間で徐冷し結晶を取り出した。

【００２３】また、同様にして、原料組成のモル比Ｌｉ／Ｔａが０．９６のタンタル酸リチウム原料を溶融して、図１に示す単結晶育成装置で単結晶を育成した。

【００２４】かくして、いずれの育成単結晶もモル比Ｌｉ／Ｔａが０．９５より大のコングルエント組成で、このときの組成変動を±０．０５％以内に抑えることができ、キュリー温度が６１０℃より高く、しかもキュリー温度のばらつきが０．２℃以下の、欠陥が無い結晶性の非常に優れたタンタル酸リチウム単結晶を育成できた。

【００２５】＜実施例２＞実施例１と同様な坩堝、単結晶育成装置、種結晶を用いて、単結晶育成炉３内のガス圧力を５００ｈＰａ±５ｈＰａに制御して結晶育成を行った。

【００２６】純度９９．９９％の炭酸リチウムと五酸化タンタルを４９．１：５０．９の割合で混合した後に電気炉で焼成し（雰囲気：大気）てモル比Ｌｉ／Ｔａが０．９７の原料６を作製した。

【００２７】作製した原料６を実施例１と同様な大きさ形状のイリジウム製の坩堝４に充填し、引き上げ方位を実施例１と同様な方位で先端部の径が６ｍｍの種結晶１０とともに、図１に示す単結晶育成装置にセットした。

【００２８】吸気口１から酸素ガス２体積％、窒素ガス９８体積％の混合ガスを吸入し圧力計８で圧力を測定しながら排気ポンプ７を排気制御して単結晶育成炉３内の圧力が５００ｈＰａ±５ｈＰａになるように制御した。

【００２９】コイル５により坩堝４を高周波加熱し、タンタル酸リチウム原料６を溶融した。原料が溶けた後、融液表面温度を融点の１６５０℃より３０～４０℃高い１６８０～１６９０℃まで昇温し、１時間保持した後、融液温度を融点まで下げた。

【００３０】種結晶１０を９．５ｒｐｍで回転させながらゆっくりと降下させ、液面に接触させた後、種結晶先端部の径が５ｍｍになるように高周波出力により液面温度を制御した。

【００３１】種結晶１０を２ｍｍ／ｈｒで回転引き上げを行いながら、徐々に温度を下げて４時間程度で結晶径１０ｃｍまで結晶頭部を育成した後に直胴部を１１ｃｍに育成させた。育成した結晶を融液から切り離した後室温まで３０時間で徐冷し結晶を取り出した。

【００３２】かくして、モル比Ｌｉ／Ｔａが０．９７のコングルエント組成で、このときの組成変動を±０．０５％以内に抑えることができ、キュリー温度が６２４℃、しかもキュリー温度のばらつきが０．２℃以下の、欠陥の無い結晶性の非常に優れたタンタル酸リチウム単結晶を育成できた。

＜実施例３＞実施例２と同様にして、原料組成をＬｉ：Ｔａ＝５０：５０（モル比１）、５ｈＰａ±１ｈＰａの酸素雰囲気下で結晶育成を行った。

【００３３】かくして、モル比Ｌｉ／Ｔａが１．０のコングルエント組成で、このときの組成変動を±０．０５％以内に抑えることができ、キュリー温度が６７５℃で、キュリー温度のばらつきが０．２℃以下の、欠陥の無い結晶性の優れたタンタル酸リチウム単結晶を育成できた。

【００３４】＜比較例＞原料組成を実施例１～３と同一

にして、大気中で結晶育成を行ったところ、組成の変動が０．１％以上で、さらにキュリー温度のバラツキが１．５℃以上もあり、原料チャージ回数が多くなるにしたがってこのバラツキが大きくなる傾向がみられた。これは天候等の原因で圧力が大きく変化し、結晶のコングルエント組成が変動したため、結晶と坩堝に残留した原料の組成にずれが生じたためと思われる。

【００３５】また、育成結晶は直胴部付近から曲がり、黒く着色していた。これは酸素分圧が２ｈＰａ程度まで低下したため酸素欠陥により黒く着色し、結晶が炉内の輻射熱を吸収し固液界面での温度勾配が低下したためと考えられる。

【００３６】

【発明の効果】以上説明したように、本発明のタンタル酸リチウム単結晶及びその製造方法によれば、欠陥が極めて少なく高品質で且つ均一組成の単結晶、すなわち、キュリー温度のばらつきがほとんど無く、結晶性に優れた単結晶を得ることができる。特に、キュリー温度が高くそのばらつきが無い単結晶を得ることができるので、非線形光学定数の大きい光学用途に好適な単結晶を提供できる。

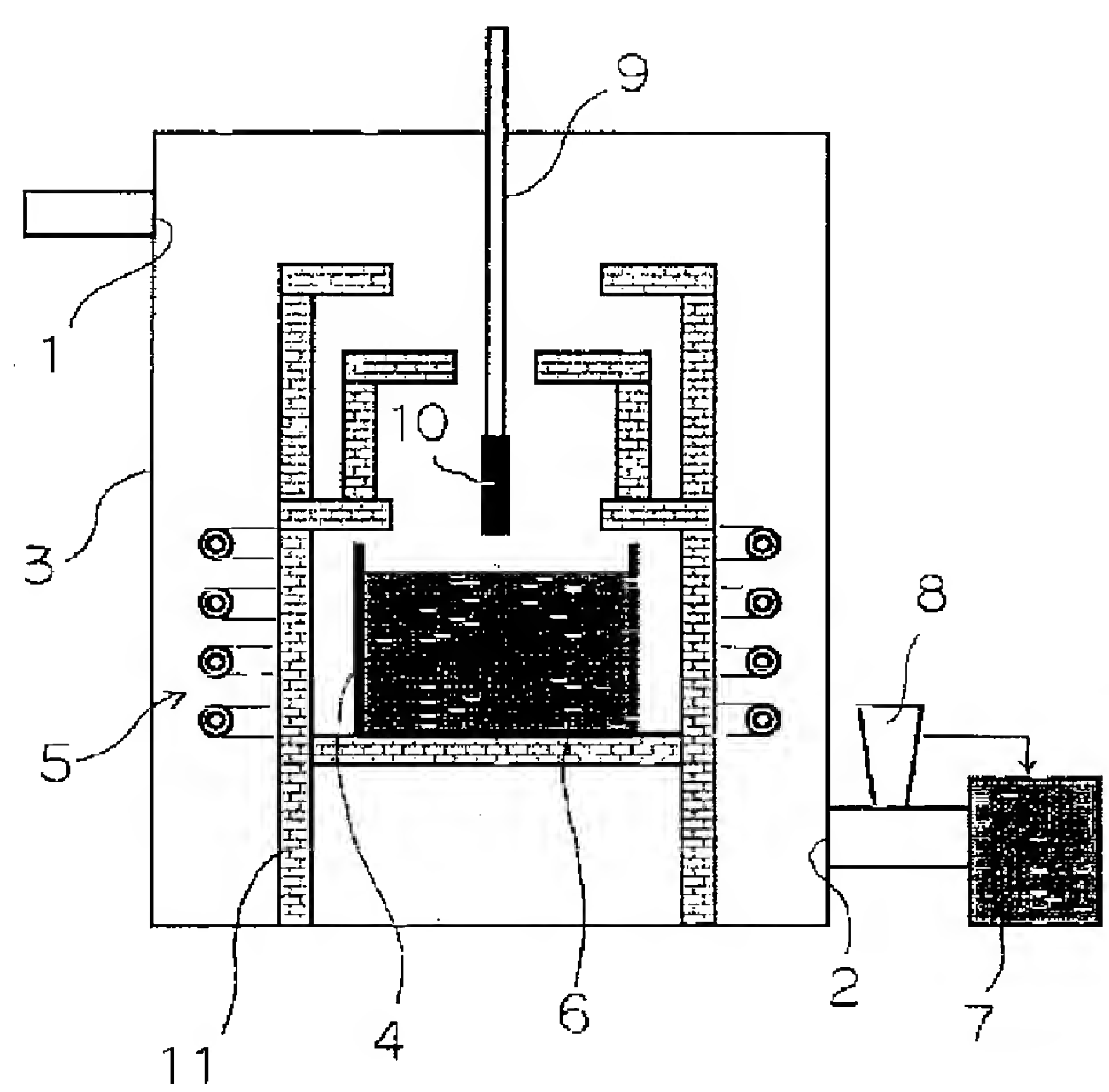
【図面の簡単な説明】

【図１】本発明に係る単結晶育成装置を模式的に説明する一部断面図である。

【符号の説明】

１：吸気口
２：排気口
３：単結晶育成炉
４：坩堝
５：コイル
６：融液
７：排気ポンプ
８：圧力計
９：支持棒
１０：種結晶
１１：耐火セラミックス

【図１】



PATENT ABSTRACTS OF JAPAN

(11)Publication number :
2003-063894

(43)Date of publication of application :
05.03.2003

(51)Int.Cl.
C30B 29/30

(21)Application number :
2001-259313
(71)Applicant :
KYOCERA CORP
(22)Date of filing :
29.08.2001
(72)Inventor :
HORI KENJI

(54) SINGLE CRYSTAL OF LITHIUM TANTALATE AND METHOD FOR PRODUCING THE SAME

(57)Abstract:
PROBLEM TO BE SOLVED: To provide the single crystal of lithium tantalate having a congruent composition close to an unconventional stoichiometric composition and having a good crystallinity, and also to provide a method for producing the same.
SOLUTION: The single crystal of lithium tantalate has a mole ratio Li/Ta of 0.95 or larger of the congruent composition and Curie temperature of 610°C or higher. The single crystal is grown in a czochralski method while the varying width of a gas pressure is being controlled below 10 hPa in a furnace for growing the single crystal where the partial pressure of an oxygen gas is 5-20 hPa and the partial pressure of an inert gas of is below 900 hPa, so that the single crystal, having few defects, a high quality and a uniform composition, can be grown.

CLAIMS

[Claim(s)]
[Claim 1]A lithium tantalate single crystal whose mole-ratio Li/Ta of a KONGURUENTO presentation is size from 0.95 and whose Curie temperature is higher than 610 **.
[Claim 2]Are a manufacturing method raised with the Czochrlski method, and, in oxygen gas partial pressure, 5-20 hPa and an inactive gas partial pressure the lithium tantalate single crystal according to claim 1 within a single-crystal-growth furnace of 900 hPa or less, A manufacturing method of a lithium tantalate single crystal pulling up a training single crystal from melt controlling the gas pressure range of fluctuation to 10 hPa or less.

[Translation done.]

* NOTICES *

JPO and INPIT are not responsible for any
damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]this invention -- as the substrate for surface acoustic wave (SAW) elements, an optical element, etc. -- suitable -- business -- **** -- a lithium tantalate single crystal -- and it is related with the manufacturing method.

[0002]
[Description of the Prior Art]Conventionally, generally the Czochrlski method is used for manufacture of a lithium tantalate single crystal. After carrying out heat melting of the raw material filled up with this method in crucible, a seed crystal is contacted to that melt, It can be made to be able to grow up to the crystal diameter made into the purpose by temperature operation, pulling up a seed crystal after an appropriate time, and the lithium tantalate single crystal of the same direction as a seed crystal can be obtained after that by temperature operation which keeps a crystal diameter constant.
[0003]. Since the melting point of lithium tantalate is about 1650 **, expensive iridium of a refractory metal is usually used as crucible material, and carry out melting of the raw material with heating of the crucible by high frequency induction heating. in order to control consumption of iridium -- an oxygen density -- as much as possible -- it is few (for example, 2 volume %) -- it is necessary to carry out Although a control atmosphere is performed by supplying the mixed gas of oxygen and inactive gas in the single-crystal-growth furnace which is a crystal training room, since the single-crystal-growth furnace exhaust port is opened wide, if no less than 30-40 hPa of outdoor air pressure is changed according to the weather, the pressure in a single-crystal-growth furnace will be changed similarly.
[0004]
[Problem(s) to be Solved by the Invention]Generally, although it is supposed under atmospheric pressure, i.e., a 1013-hPa condition, that it is the KONGURUENTO presentation of a lithium tantalate single crystal about Li:Ta=48.3:51.7, If a pressure falls by about 40 hPa, a KONGURUENTO presentation will be changed to about Li:Ta=48.5:51.5.
[0005]Thus, since the raw material presentation which a presentation gap of a raw material and a training crystal not only occurs, but remains in crucible since a KONGURUENTO presentation will be changed if the pressure in a single-crystal-growth furnace is changed will also shift, if continuously filled up with a raw material, a big presentation gap will arise. In order to prevent this, adjustment of a raw material presentation and exchange of a raw material are needed each time. Since the KONGURUENTO presentation under atmospheric pressure has little content of Li, there are many Li defects under crystal, crystallinity falls, and it is not preferred as substrates or the objects for optical elements, such as electronic parts.
[0006]So, it aims at having the KONGURUENTO presentation near the stoichiometric composition which is not in the former, and providing a good crystalline lithium tantalate single crystal and a manufacturing method for the same in this invention.
[0007]
[Means for Solving the Problem]In order to solve an aforementioned problem, mole-ratio Li/Ta of a KONGURUENTO presentation is size from 0.95, and a lithium tantalate single crystal of this invention is characterized by Curie temperature being higher than 610 **.
[0008]A manufacturing method of the above-mentioned lithium tantalate single crystal, It was a manufacturing method which raises a single crystal with the Czochrlski method, and oxygen gas partial pressure pulled up a training single crystal from melt, while 5-20 hPa and an inactive gas partial pressure controlled the gas pressure range of fluctuation to 10 hPa or less within a single-crystal-growth furnace of 900 hPa or less.
[0009]A defect thereby always becomes possible [few things for which it is quality and a single crystal of uniform composition is raised continuously].

[0010]
[Embodiment of the Invention]Below, the embodiment of this invention is described in detail based on the drawing which expressed typically.
[0011]Drawing 1 is a sectional view of the high-frequency-induction-heating-type single crystal growth device used by this invention.
[0012]First, where the crucible 4 made from iridium surrounded with the fire-resistant ceramics 11 allocated cylindrical in the single-crystal-growth furnace 3 provided with the inlet port 1 and the exhaust port 2 of gas is filled up with the lithium tantalate raw material of a KONGURUENTO presentation, The mixed gas of oxygen (0.5 to 2 volume %) and inactive gas (they are nitrogen or argon 98 to 99.5 volume %) is inhaled at the single-crystal-growth furnace 3 from the inlet port 1.
[0013]Next, control the exhaust air pump 7 by the pressure gauge 8 formed near the exhaust port 2, measuring the pressure in the single-crystal-growth furnace 3, and, in oxygen gas partial pressure, 5-20 hPa and an inactive gas partial pressure within a single-crystal-growth furnace of 900 hPa or less, Controlling the gas pressure range of fluctuation to 10 hPa or less, within a single-crystal-growth furnace, it controls so that the gas pressure range of fluctuation becomes the constant pressure of 10 hPa or less.
[0014]And with the coil 5 provided in the circumference of the crucible 4, high frequency induction heating is performed to the crucible 4, the raw material in the crucible 4 is fused, and it is considered as the melt 6. Then, contacting the seed crystal 10 of the predetermined direction established at the tip of the bearing bar 9 to the melt 6, controlling a high frequency output, and controlling cooking temperature, pull up the bearing bar 9, it is made to rotate, and a crystal is grown up.
[0015]In oxygen gas partial pressure, in the above-mentioned manufacture, 5-20 hPa and an inactive gas partial pressure within a single-crystal-growth furnace of 900 hPa or less, The reason for raising a single crystal, while controlling the gas pressure range of fluctuation to 10 hPa or less, the KONGURUENTO presentation generally known as raising a single crystal in a gas atmosphere of 900 hPa or less -- Li -- it is because it became clear that it was necessary to control pressure fluctuation to 10 hPa or less in order for it to become clear that it becomes rich and to control a composition change within **0.05%.
[0016]By the above, a defect is quality very small, and from 0.95, it is size, and Curie temperature is higher than 610 **, and the single crystal of uniform composition, i.e., mole-ratio Li/Ta of a KONGURUENTO presentation, can obtain the outstanding single crystal which does not almost have dispersion in Curie temperature. Since Curie temperature is high and there is no dispersion, it is applicable to the outstanding optical application with a large nonlinear optics constant.
[0017]
[Example]Next, the more concrete example of this invention is described.
[0018]After mixing the raw material of tantalum pentoxide with lithium carbonate of 99.99% of <Example 1> purity at a rate of 48.8:51.2, it calcinated with the electric furnace (atmosphere: atmosphere), and mole-ratio Li/Ta of the raw material presentation produced the lithium tantalate raw material of 0.953.
[0019]It set in the high-frequency-induction-heating type single crystal growth device shown in drawing 1 with the seed crystal 10 whose path of a tip part is 6 mm in the direction which filled up the crucible 4 made from iridium 180 mm in inside diameter, and 180 mm in height with the produced raw material, raised it, and leaned 34 degrees - 44 degrees of directions to Z shaft orientations from the Y-axis.
[0020]Then, the mixed gas of oxygen gas 1 volume % and nitrogen gas 99 volume % was inhaled from the inlet port 1, exhaust control of the exhaust air pump 7 was performed, measuring gas pressure with the pressure gauge 8, and it controlled so that the gas pressure in the single-crystal-growth furnace 3 was set to 900hPa**5hPa.
[0021]Subsequently, the coil 5 performed high frequency induction heating to the crucible 4, and the lithium tantalate raw material was fused. After carrying out temperature up of the melt skin temperature after a raw material melts and holding it for 1 hour to 1680-1690 ** higher 30-40 ** than 1650 ** of the melting point, melt temperature was lowered to the melting point.
[0022]Subsequently, after making it descend slowly and making an oil level contact,

rotating the seed crystal 10 at 9.5 rpm, oil-level temperature was controlled by the high frequency output so that the path of a seed crystal tip part was set to 5 mm. And it pulls up rotating the seed crystal 10 by 2 mm/hr, and after lowering temperature gradually and making a crystal head raise to the crystal diameter of 10 cm in about 4 hours, the body part was made to raise to 11 cm. Thus, after separating the raised crystal from melt, it cooled slowly to the room temperature in 30 hours, and the crystal was taken out.
[0023]Similarly, mole-ratio Li/Ta of the raw material presentation fused the lithium tantalate raw material of 0.96, and raised the single crystal with the single crystal growth device shown in drawing 1.
[0024]Mole-ratio Li/Ta from 0.95 any training single crystal in this way by adult KONGURUENTO presentation. The composition change at this time could be suppressed within **0.05%, Curie temperature was higher than 610 **, and the dramatically outstanding lithium tantalate single crystal of the crystallinity in which dispersion in Curie temperature moreover does not have a defect of 0.2 ** or less has been raised.
[0025]Using the same crucible as <Example 2> example 1, the single crystal growth device, and the seed crystal, the gas pressure in the single-crystal-growth furnace 3 was controlled to 500hPa**5hPa, and crystal training was performed.
[0026]After mixing tantalum pentoxide with lithium carbonate of 99.99% of purity at a rate of 49.1:50.9, it calcinated with the electric furnace and ** (atmosphere: atmosphere) mole-ratio Li/Ta produced the raw material 6 of 0.97.
[0027]It set in the single crystal growth device which fills up the crucible 4 made from iridium of the same size shape as Example 1 with the produced raw material 6, raises it, and shows drawing 1 a direction with the seed crystal 10 whose path of a tip part is 6 mm in the same direction as Example 1.
[0028]Having inhaled the mixed gas of oxygen gas 2 volume % and nitrogen gas 98 volume % from the inlet port 1, and measuring a pressure with the pressure gauge 8, it controlled so that exhaust control of the exhaust air pump 7 was carried out and the pressure in the single-crystal-growth furnace 3 was set to 500hPa**5hPa.
[0029]High frequency induction heating of the crucible 4 was carried out with the coil 5, and the lithium tantalate raw material 6 was fused. After carrying out temperature up of the melt skin temperature after a raw material melts and holding it for 1 hour to 1680-1690 ** higher 30-40 ** than 1650 ** of the melting point, melt temperature was lowered to the melting point.
[0030]After making it descend slowly and making an oil level contact, rotating the seed crystal 10 at 9.5 rpm, oil-level temperature was controlled by the high frequency output so that the path of a seed crystal tip part was set to 5 mm.
[0031]After lowering temperature gradually and raising a crystal head to the crystal diameter of 10 cm in about 4 hours, 11 cm was made to raise a body part, performing rotation raising for the seed crystal 10 by 2 mm/hr. After separating the raised crystal from melt, it cooled slowly to the room temperature in 30 hours, and the crystal was taken out.
[0032]Mole-ratio Li/Ta can suppress the composition change at this time within **0.05% by the KONGURUENTO presentation of 0.97 in this way, Curie temperature has raised the dramatically outstanding lithium tantalate single crystal of the crystallinity in which dispersion in Curie temperature moreover does not have a 624 ** defect of 0.2 ** or less.
Crystal training was performed for the raw material presentation under Li:Ta=50:50 (mole ratio 1) and 5hPa**1hPa oxygen environment like <Example 3> example 2.
[0033]In this way, mole-ratio Li/Ta could suppress the composition change at this time within **0.05% by the KONGURUENTO presentation of 1.0, and Curie temperature has raised the lithium tantalate single crystal which excelled [** / 675] in the crystallinity as for which dispersion in Curie temperature does not have a defect of 0.2 ** or less.
[0034]When the <comparative example> raw material presentation was made the same as that of Examples 1-3 and crystal training was performed in the atmosphere, change of the presentation was 0.1% or more, there were not less than 1.5 ** of variations of Curie temperature further, and the tendency for this variation to become large was seen as the number of times of raw material charge increased. A pressure changes a lot by causes, such as the weather, and since the gap arose in the presentation of the raw material which remained to a crystal and crucible since the KONGURUENTO presentation of the crystal was changed, this is considered.

[0035]It turned at the training crystal from near a body part, and it was colored black. Since oxygen tension fell to about 2 hPa, an oxygen deficiency colors black, and since the crystal absorbed the radiant heat in a furnace and the temperature gradient in the solid-liquid interface fell, this is considered.
[0036]
[Effect of the Invention]As explained above, according to a lithium tantalate single crystal of this invention, and a manufacturing method for the same, a defect is quality very small, and there is almost no single crystal of uniform composition, i.e., dispersion of Curie temperature, and the single crystal excellent in crystallinity can be obtained. Since the single crystal which Curie temperature is high and does not have the dispersion in particular can be obtained, the suitable single crystal for an optical application with a large nonlinear optics constant can be provided.

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1]the single crystal growth device concerning this invention is explained typically -- it is a sectional view in part.
[Description of Notations]
1: Inlet port
2: Exhaust port
3: Single-crystal-growth furnace
4: Crucible
5: Coil
6: Melt
7: Exhaust air pump
8: Pressure gauge
9: Bearing bar
10: Seed crystal
11: Fire-resistant ceramics

[Translation done.]
* NOTICES *

JPO and INPIT are not responsible for any
damages caused by the use of this translation.

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DRAWINGS

[Drawing 1]

[Translation done.]